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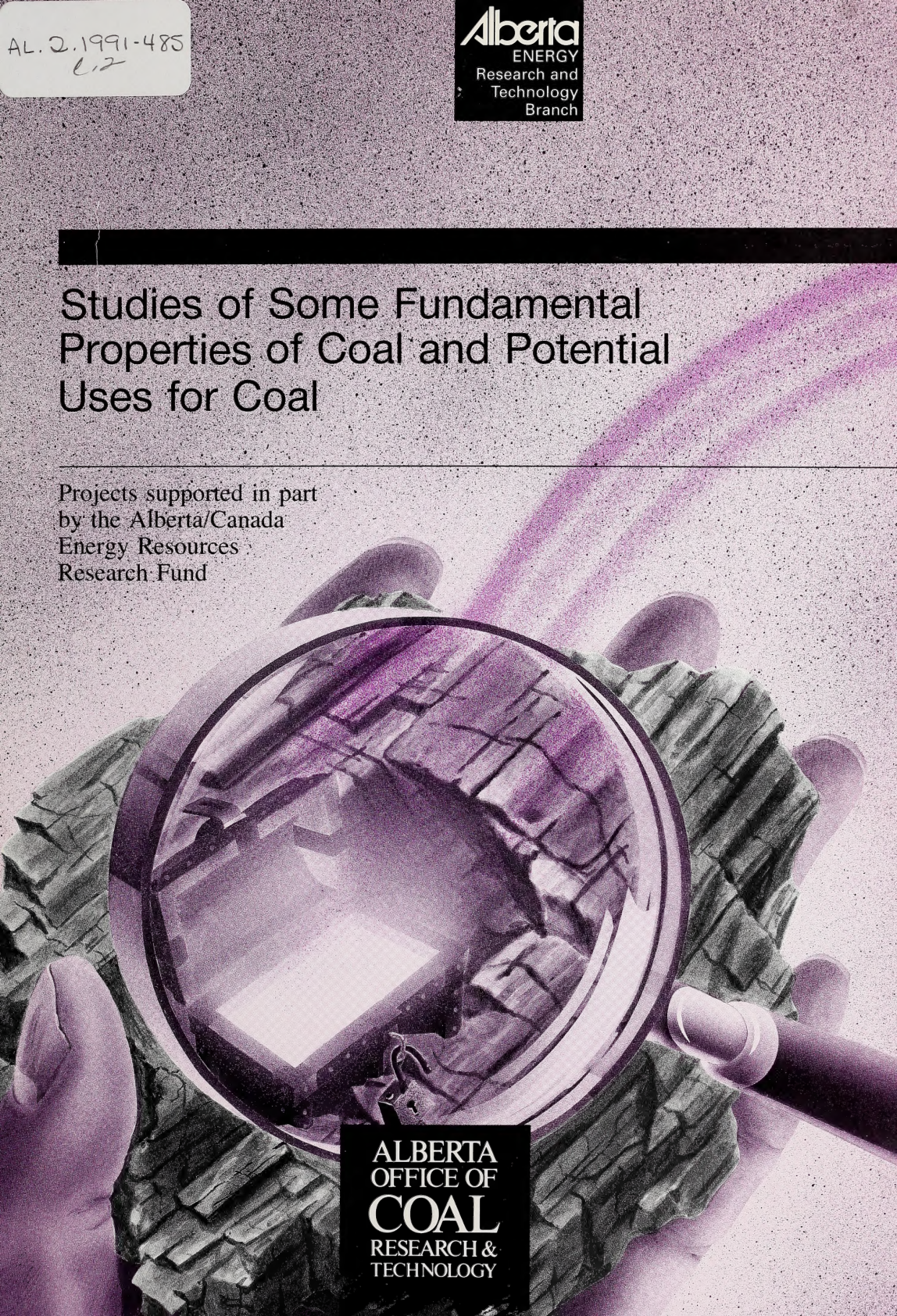
Studies of Some Fundamental Properties of Coal and Potential Uses for Coal

Projects supported in part
by the Alberta/Canada
Energy Resources
Research Fund



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Foreword

Beginning in 1976, numerous projects were initiated in Alberta by industry and academic research institutions to help make better use of Alberta's energy resources.

These research, development and demonstration efforts were funded by the Alberta/Canada Energy Resources Research Fund (A/CERRF), which was established as a result of the 1974 agreement on oil prices between the federal government and the producing provinces.

Responsibility for applying and administering the fund rested with the A/CERRF Committee, made up of senior Alberta and federal government officials.

A/CERRF program priorities focused primarily on coal, energy conservation and renewable energy, and conventional energy resources. In 1988/89, a hydrogen research component was added.

Administration for the A/CERRF program was provided by staff within the Research and Technology Branch of Alberta Energy.

In recognition of the importance of coal to Alberta's economy, the Alberta Office of Coal Research and Technology was established in 1984 within Alberta Energy and Natural Resources (now Alberta Energy). Its primary purpose is to encourage the development and application of new technologies related to Alberta coals. The Office provides funding contributions to research and development projects in industry, academic institutions and other research establishments. It also monitors the progress of these projects in an overall program of improving the production, transportation and marketability of Alberta coals.

The A/CERRF program ended on March 31, 1991 and the support it provided has been replaced by funding from the Alberta government.

A series of technology transfer booklets begun in 1986 with A/CERRF support continues to make research results available to industry and others who can use the information.

This service will continue until all A/CERRF projects have been described.

For more information about other publications in the series, please refer to page 11.

Studies of Some Fundamental Properties of Coal and Potential Uses for Coal

Much of the science in coal research involves making empirical observations of the "cause-and-effect" variety. There is nothing wrong with this approach, and it is popular because such observations are often all that is required for progress to be made. Nonetheless, some phenomena are less reluctant than others to yield their secrets. This is where more drastic measures are required, where scientists must try peering into the molecular and atomic structures of substances to gain the needed information.

Thus, the coal research program which has been carried out in Alberta since the early 1980s contained several projects that were primarily concerned with some fundamental properties of coal. These projects were conducted in parallel with the other, more empirical projects and served to support the latter group. They are reported here.

Associated with these studies of fundamental properties was a second group of projects concerned either with potential new markets for Alberta coals, or the re-defining of old markets which are still comparatively small at present but have some promise for growth.

All these projects were funded by the Alberta/Canada Energy Resources Research Fund (A/CERRF), as administered by the Alberta Office of Coal Research and Technology.

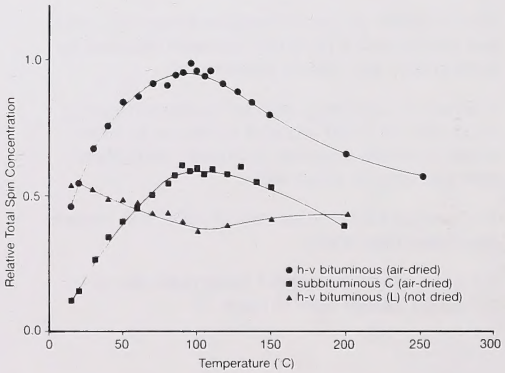
Magnetic and Electric Properties of Alberta Coals

In this project, carried out at The University of Calgary, sophisticated analytical procedures were used to investigate bituminous and subbituminous Alberta coals on a molecular scale. Information about the electronic structure of atoms and molecules, and the bonds formed between various components of coal, was obtained by Continuous Wave Electron Paramagnetic Resonance (CW-EPR). The objective was to determine the role that coal rank, mineral content, maceral content and water content play in establishing the susceptibility of coals to spontaneous combustion.

This investigation was also expected to provide useful insights about free radical reactions that occur when coal is converted to liquid hydrocarbons.

A unique sample holder was designed and constructed for this study. It allowed dynamic, in situ CW-EPR studies of coals to be performed for the first time over the temperature range of 20° to 600°C. Also developed for this project were a precise temperature controller, software that enabled the 9 GHz CW-EPR spectrometer to be operated under computer control, and an on-line display of information derived from the CW-EPR spectrum.

Oxidation and Water-Removal Behaviour of High-Volatile Bituminous and Subbituminous Coals



(Source: Magnetic and Electric Properties of Alberta Coals, Buckmaster, H.A., The University of Calgary, 1989.)

Examination of coal samples exposed to dry or moist air or nitrogen, while the temperature was varied and controlled, showed that oxidation and the removal of weakly associated water occurred differently in the bituminous and subbituminous coals. The spin concentration measured by CW-EPR increased by a factor of six in the subbituminous coal, while it increased by only a factor of two in the bituminous coal. This behaviour was noted over the temperature range of 20° to 100°C. These results confirmed that subbituminous coals are more susceptible to spontaneous combustion than bituminous coals, and the presence of oxygen is necessary for the formation of free radicals.

Similarly, an investigation of the presence of cobalt and manganese in coal showed they play an important role in making coal more susceptible to spontaneous combustion. Also, the moisture content of a high-volatile bituminous coal affected the formation of free radicals, and the vitrinite maceral also encouraged free radical formation.

One especially interesting discovery was the fact that two free radical species were present in all coal samples. Apparently, changes in the concentration of one of these species are responsible for the observed increases in spin concentration. This is related to the susceptibility of coal to undergo spontaneous combustion.

The researchers drew the following conclusions from their observations:

- the heating of subbituminous coals in the presence of dry air produces more free radicals than when bituminous coals are treated in the same manner;
- the presence of minerals, such as cobalt and manganese, plays a significant role in producing free radicals when coals are heated in the presence of dry air;
- the number of free radicals created when dry or moisture-saturated coals are heated in the presence of dry air is determined primarily by the vitrinite maceral content of the coal; and
- the free radical content attains a maximum level at approximately 100°C.

Distributed Chemical and Physical Properties of Coal

Depending on the purpose, analytical investigations of coal can be divided into two groups: (1) those that attempt to extract minute details about particular components and properties; and (2) those that aim to relate generalized properties of coal to certain behavioural characteristics to provide a simple method of making predictions about those characteristics. The latter group is represented in a project carried out at The University of Calgary.

The premise of this project was that correlations between measured properties of coal and their behaviour are poor and they are caused by the unavoidable heterogeneity of coal. Thus, it is impossible to guarantee that samples of coal used for one type of analysis are identical in all respects to those used to assess a particular processing capability.

An alternative approach is to assume that chemical and physical properties of coal are spatially distributed, and a statistical analysis of these distributions can lead to predictions about coal behaviour in, for example, combustion, liquefaction or gasification processes. Furthermore, it is believed that these distributions, whether on a macro scale (such as in a coal seam) or a molecular scale, are not independent of each other.

In this project, a branch of mathematics called fractal analysis was used to derive relationships between underlying distributions of coal attributes and observed chemical or physical properties.

Following a literature review on the subject, an examination was begun of coal-derived liquids. Asphaltene content and molecular weight distributions were tracked and compared with other chemical and physical properties. Also, the adsorption of various gases on coal was studied as a potential method for establishing correlations between coal characteristics and processing behaviour.

Tentative correlations were established between processing behaviour and coal rank, as well as with the chemical and physical heterogeneity of the coal.

A possible measure of chemical heterogeneity is a fractal dimension arising from the low coverage adsorption of a single vapour on a coal. Physical heterogeneity might be measured by a second fractal dimension arising from the variation in the surface area affected by several vapours as they adsorb onto the coal surface.

Sulphur Isotope Studies of Coal

A better understanding of the influence of sulphur contained in coal is important in various coal conversion processes, ranging from combustion, to liquefaction, to gasification. Furthermore, the role of sulphur in these reactions is complicated by the fact that both organic and inorganic forms of the element can occur simultaneously.

Thus, there is a need to develop a relatively simple and inexpensive apparatus for determining different forms of sulphur in coal. One approach being pursued by researchers at The University of Calgary involves using stable isotope analyses of different forms of sulphur. This not only provides information about the geochemistry of fossil fuels, but also permits tracking of the different forms of sulphur as they undergo conversion in various chemical processes.

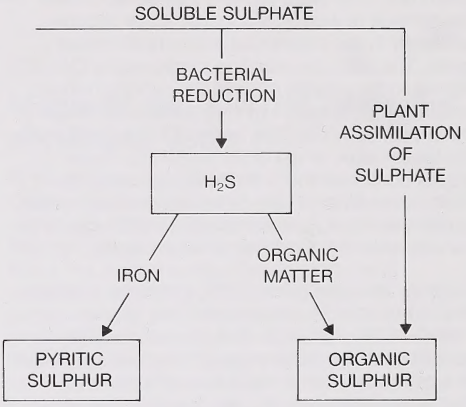
In previous work at The University of Calgary, researchers developed a non-isothermal, pyrolysis-stable isotope technique to study forms of sulphur (and other elements) in fossil fuels. The technique involves heating a substance in a quartz reactor to progressively higher temperatures and analysing the evolved gases with continuous nitrogen purging. For sulphur studies, the evolved gas is hydrogen sulphide (H_2S).

In this project, coals from several Canadian sources — including low-sulphur Alberta coal and high-sulphur coal from Nova Scotia — were analysed by the pyrolysis-stable isotope technique and other methods, including Kiba Extraction which was claimed to be capable of distinguishing between organic and inorganic forms of sulphur. A critical assessment of the Kiba technique using sulphur isotope labelling revealed that this claim was overly optimistic.

After a more cost-effective quartz reactor was designed and a computer-based temperature controller developed, several samples of a control coal containing two per cent of sulphur by weight were pyrolysed to ascertain the reproducibility of the method. It was found that reproducibility was variable, apparently because of sample inhomogeneity. H_2S recovery was improved, however, by using flush gases containing hydrogen. They included water, methane and a mixture of helium and hydrogen. The helium/hydrogen mixture subsequently became the standard flush gas for the remaining experiments.

Having refined the experimental technique, the researchers applied it to a suite of low-sulphur coals and peat. It was found that coal samples

Methods of Sulphur Incorporation in Organic-Rich Sediments



(Source: Isotopic Studies of Sulphur in Coal, Krouse, H.R. and C.J. Yonge, The University of Calgary, 1989.)

containing minimal amounts of inorganic sulphur showed differences in H_2S evolution which could be related to coal rank. This behaviour in the sulphur isotope data can be interpreted in terms of the relative stabilities of various organic sulphur structures, but the technique is not sufficiently sophisticated at present to pinpoint which sulphur-bearing functional groups are responsible for the observed data.

Following the completion of this project, two state-of-the-art computerized stable isotope mass spectrometers were installed at The University of Calgary. This should further improve the capabilities of this promising analytical technique since much smaller samples can be analysed. The technique should be particularly useful for studying desulphurization schemes, including bacterial oxidation of organic sulphur.

Distribution of Oxygen Forms in Western Canadian Low-Rank Coals

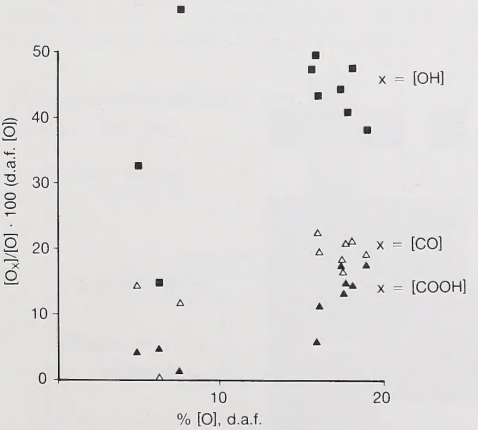
It is generally conceded by coal scientists that the chemical properties and physical behaviour of low-rank coals are largely determined by the reactive oxygen content, which can account for 75 to 90 per cent of the total oxygen content. Also, this reactive oxygen is usually found in phenolic hydroxyl (-OH), carboxyl (-COOH) and carbonyl (=CO) groups, while unreactive oxygen exists mostly in heterocyclic combinations.

However, there is considerable uncertainty about the relative abundances of individual reactive oxygen groups in low-rank coals. Therefore, the objective of a project carried out at the University of Alberta was to measure the concentrations and thermal stabilities of the dominant oxygen forms in a suite of Alberta bituminous and subbituminous coals. It was anticipated that this investigation might reveal an explanation for the dissimilar behaviour of coals that appear outwardly to be similar. For example, it is known that coals with virtually identical elemental and petrographic compositions will sometimes display very different pyrolytic behaviour and respond very differently to hydrogenation and solubilization in liquefaction schemes.

The concentration of reactive oxygen groups in each type of coal was determined by conventional "wet chemistry" methods, and the results were expressed as concentration of oxygen in each group, as weight percentages of "daf" coal and also as fractions of the total oxygen content of the coal.

Although earlier work showed that little -OH or =CO loss could be expected below 400°C, kinetic considerations indicated that loss of -COOH should be more significant. Indeed, it was found that some of the coals experienced a 40 to 60 per cent loss of -COOH concentration at 300°C, but others lost only 15 to 20 per cent. Substantial carboxyl group losses occurred when coals were "flashed" at 300°C; up to 85 per cent in some cases.

Variable Distribution of Oxygen-Bearing Functional Groups in "Similar" Low-Rank Coals



(Source: Berkowitz, N. Distribution of Oxygen Forms in Alberta Low Rank Coals, University of Alberta, 1990.)

Qualitatively, Alberta coals exhibited characteristics similar to other coals, but some differences did emerge. For example, they appear to contain significantly higher levels of phenolic -OH. This might explain their low tar yields when pyrolyzed. Also, from plots of oxygen species as a fraction of total oxygen, it is evident that coals appearing otherwise to be similar have significantly different chemistry.

It was also shown that a substantial portion of the total oxygen content of low-rank coals can be removed by low-temperature pyrolytic pre-treatment. This may reduce the requirement for external hydrogen in coal liquefaction processes.

Geostatistics

Geostatistics has been used successfully in the mineral industry to assess ore bodies accurately by analysing the spatial correlations present in exploration data. Also, the technique has been successfully applied to the evaluation of coal deposits where the results of analysis enabled improved mine planning and enhanced the quality of coal production.

Thus, the objective of a project carried out by the Coal Mining Research Company was to develop the capability of analysing coal quality variation at the deposit level. It was reasoned that knowledge obtained through geostatistical analysis might enable better control of the quality and consistency of mined coal. This could enhance the efficiency of coal preparation plants.

During the term of the project, one professional from the Coal Mining Research Company was trained in the theory and applications of geostatistics so as to enhance the services of the company in identifying and developing applications in coal mining and preparation.

Electrolysis of Coal Slurries

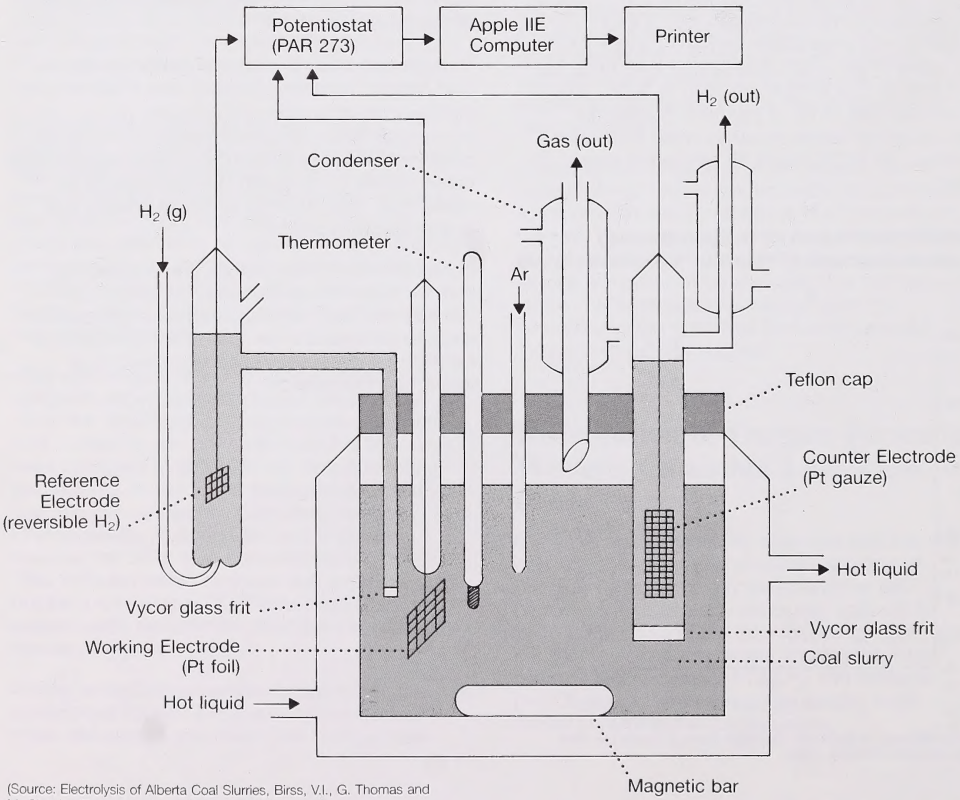
Research studies in several laboratories have shown that hydrogen is readily and inexpensively produced when acidic coal slurries are electrochemically oxidized. The principal advantage of this method for producing hydrogen stems from the relatively lower voltage required to liberate hydrogen, as opposed to the conventional method involving the electrolysis of water. Furthermore, electrochemical oxidation of acidic coal slurries can produce other gases of commercial importance in a pure state, such as carbon monoxide and carbon dioxide.

Because most studies of this kind have been carried out in the United States using U.S. coal, a project was initiated at The University of Calgary to determine the feasibility of using electrochemical methods to oxidize Alberta coals and recover products having commercial value. An additional objective was to determine whether correlations exist between oxidation rates and coal rank or origin.

During the initial stage of this project, coals originating from four mines (Highvale, Coal Valley, Vesta and Bow City) were pulverized and slurried in a mixture of sulphuric acid and ferrous sulphate. The slurries were exposed to an electric current at room temperature to produce hydrogen and various oxidized coal products. The experiments also included procedures to regenerate the oxidized surfaces of the coal particles to encourage additional reaction. In later work, experiments were carried out at 90°C to obtain increased reactivity. Besides hydrogen, the other principal product obtained from these experiments was carbon dioxide.

A second order kinetic analysis of the electrochemical data produced rate constants for the process. The latter correlate well with the aromaticity and fixed

Schematic Diagram of Electrochemical Cell Used in Electrolysis of Coal Slurries.



(Source: Electrolysis of Alberta Coal Slurries, Birss, V.I., G. Thomas and M. Chettiar, The University of Calgary, 1988.)

carbon content of the various coals. In decreasing order, the electrochemical activity was found to be Highvale, Coal Valley, Vesta and Bow City.

This research work resulted in several new methods for regenerating the coal surface to allow high reaction rates to occur over oxidation periods extending up to 25 hours.

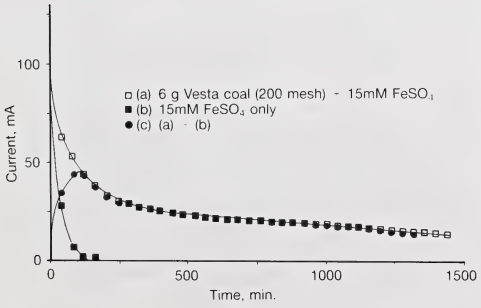
Electrochemical oxidation was also carried out in acetic acid slurries. Higher reaction rates and concentrations of solution-soluble low molecular weight organic compounds were observed, and less carbon dioxide was produced than in sulphuric acid slurries. Also, the use of certain chemicals in place of ferrous sulphate to mediate the coal oxidation reaction resulted in rate constants that were 100 times higher than with iron.

The results using acetic acid were sufficiently promising that a second project was initiated, this time using the following media: aqueous organic solutions of acetic, formic and oxalic acids; aqueous alkaline solutions having a pH of 8-12; and aqueous/non-aqueous solvent mixtures, all containing various reduction/oxidation (redox) mediators. The purpose in using these new reaction media was to provide product selectivity and high reaction rates. It was thought this could represent an important step toward a new process for liquefying coal.

It was learned that oxalic acid is not suitable for coal oxidation, but experiments using aqueous/non-aqueous mixtures and alkaline aqueous media showed promise.

Organic solvents that included dimethylsulphoxide, acetonitrile and tetrahydrofuran mixed with sulphuric acid provided good conductivity and maintained satisfactory reaction kinetics of the $Fe^{+2/+3}$ mediator. A $Co^{+2/+3}$ bipyridyl complex was found to be a suitable mediator in alkaline slurries.

I/t Response Obtained at 90°C and 1.0 V in 1 M H_2SO_4



(Source: Electrolysis of Alberta Coal Slurries, Birss, V.I., G. Thomas and M. Chettiar, The University of Calgary, 1988.)

In most of these reactions, the electrochemical activity of the coal remained high, even after long oxidation periods, and comparatively little CO_2 was produced. This indicated a high extent of formation of liquid organic products.

Activated Carbon from Coal

As Canada's population grows, the demand for clean water and the activated carbon used in water purification are expected to rise.

In producing activated carbon from coal, manufacturers tend to use proprietary processes designed to work best with individual types of coal. However, in projects carried out at The University of Calgary, the objective was to design an activated carbon reactor capable of handling a variety of coals. This was to be achieved by providing methods to control many of the process variables and conditions. An additional objective was to publicize the operating conditions so as to encourage more manufacturers to enter the field.

During initial experiments from 1980 to 1983, a small, fixed-bed reactor was used to screen a number of Alberta subbituminous coals for their suitability as feedstocks for activated coal. Approximately 195 experimental runs were made during that two-year period. This work identified three promising coals which were studied in detail during a second project. Before this latter project could begin, a special reactor was designed and built.

The reactor comprised two slightly inclined, cylindrical chambers that were kept separate from each other. A preheat chamber, measuring 122 cm (4 ft.) long and having an internal diameter of 21 cm (8 in.) was fitted with a hollow, motor-driven shaft to which paddles were attached. The rotating action of the shaft and the shape of the paddles caused coal to move through the chamber while heat was supplied by a propane burner located at one end of the unit. In the preheat chamber, all the coal moisture and most of the volatile matter were removed during typical residence times of one hour at approximately 650°C.

The second chamber, which was used for coal activation, comprised two sections identical to the preheat chamber which were bolted together. A paddle arrangement was used here as well. Two propane burners supplied the required heat. Steam and flue gas were injected into the chamber, and a provision was made to introduce tertiary air through six ports in each of the activation sections. In this chamber, the micropore structure of the coal was developed by controlling reaction conditions such as temperature, gas pressure and interactions between steam and carbon.

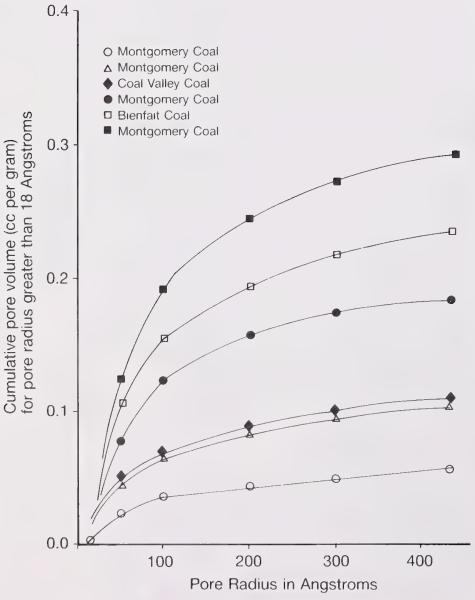
The kiln was used to complete 14 semi-continuous production runs using Coal Valley high-volatile bituminous and Montgomery subbituminous coals from Alberta, as well as Bienfait lignite coal from Saskatchewan. The residence time in the activation section of the reactor ranged from two to six hours, at temperatures that varied from 595 to 834°C.

The following physical properties were obtained for some selected activated coals:

	Bienfait	Montgomery	Coal Valley
Total Surface Area, m^2/g	511	510	345
with pore radius <18A	416[1]	403[1]	293[1]
with pore radius >18A	95[1]	107[1]	52[1]
Iodine No.	478	533	436
Phenol No.	62	56	56
Methylene Blue No.	64	80	33
Ash content, per cent	21.9	31.8	19.7
Yield, per cent dry basis	48.3	42.3	50.3
Bulk Density, g/cc	0.495	0.486	0.488

[1]Calculated values

Pore Size Distribution of Activated Carbon by Mercury Penetration



(Source: Preparation of Activated Carbon from Coal, Chowdhury, A.I., E.S. Hall and E.L. Tollefson, The University of Calgary, 1988.)

It was concluded that the Bienfait lignite and Montgomery subbituminous coals were readily activated to products with qualities in the low end of the commercial range of activated coals. Higher temperatures than were possible with this particular reactor would be needed to produce suitable activated carbon from Coal Valley high-volatile coal.

The University of Calgary researchers believe these results demonstrated that subbituminous coals from western Canada show considerable promise for production of activated carbon, and they recommended that an economic study of commercial-scale production should be undertaken.

In 1983, a stable isotope research study funded by A/CERRF was undertaken by researchers at The University of Calgary. The objective of this investigation was to demonstrate that isotopic measurements of the natural gas found in Alberta's various gas fields could provide important information about the origin and characteristics of this resource.

Thus, carbon and hydrogen isotope data were obtained for methane, ethane and propane samples from Alberta wells, which ranged in depth to 4 000 m. These data covered dry biogenic gas (resulting from the decomposition of living organisms) from shallow wells, to oil-associated gas from the Devonian Formation, to dry, thermal gas found in the Deep Basin and formed by thermal cracking of petroleum. During gas exploration, this data base can be helpful in testing relationships among isotopic, physical, chemical and geological characteristics.

In particular, the availability of the carbon isotope data base proved useful in demonstrating that sour (H_2S-rich) gas could be generated by thermochemical reduction of sulphate with light hydrocarbon gases.

While the study revealed interesting information about the origins of natural gas, and showed a marked difference between gas found above 1 000 m and that found below this level, the investigation also allowed the establishment and development of a state-of-the-art analytical facility at The University of Calgary. The capabilities of this laboratory are being used increasingly by Alberta resource companies to solve problems, such as locating ruptures in oil wells, identifying sources of gas leakage into basements, and monitoring in situ recovery operations in oil sands.

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Contacts

Detailed information about the magnetic and electric properties of Alberta coals may be obtained from:

Dr. H.A. Buckmaster
Professor of Physics
Department of Physics and Astronomy
The University of Calgary
2500 University Drive N.W.
Calgary, Alberta
T2N 1N4

Telephone: (403) 220-5385

For more information about the distributed chemical and physical properties of Alberta coals, contact:

Dr. P.J. Crickmore
Associate Professor
Department of Chemical Engineering
536 Chemical-Mineral Engineering Building
University of Alberta
Edmonton, Alberta
T6G 2G6

Telephone: (403) 492-3321

Further information regarding sulphur isotope studies of Alberta coals, and isotopic compositions of fossil fuels generally, is available from:

Dr. H.R. Krouse
Professor
Department of Physics and Astronomy
The University of Calgary
2500 University Drive N.W.
Calgary, Alberta
T2N 1N4

Telephone: (403) 220-5385

For additional information regarding the distribution of oxygen forms in low-rank Alberta coals, contact:

Dr. N. Berkowitz
Emeritus Professor of Fuel Science
Department of Mining, Metallurgical and Petroleum Engineering
606 Chemical-Mineral Engineering Building
University of Alberta
Edmonton, Alberta
T6G 2G6

Telephone: (403) 492-3337

Details about the electrolysis of coal slurries are available from:

Dr. V.I. Birss
Associate Professor of Chemistry
Department of Chemistry
The University of Calgary
2500 University Drive N.W.
Calgary, Alberta
T2N 1N4

Telephone: (403) 220-5341

More information about the production of activated carbon from Alberta coals is available from:

Dr. E.L. Tollefson
Professor
Department of Chemical and Petroleum Engineering
The University of Calgary
2500 University Drive N.W.
Calgary, Alberta
T2N 1N4

Telephone: (403) 220-5751

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Chairman
Alberta Office of Coal Research and Technology
Alberta Energy
11th Floor, North Petroleum Plaza
9945 - 108 Street
Edmonton, Alberta
T5K 2G6

Telephone: (403) 427-8042
Telex: 037-3676
Fax: (403) 422-0975

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